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(19) (CA) **APPLICATION FOR CANADIAN PATENT** (12)

(54) High Pressure Hot Separator

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ABSTRACT

A high pressure hot separator for the separation of a head product into a gas/vapour phase and a sump product is disclosed. The head product is produced in a process of the high pressure hydration of coal, tar, mineral oils, their distillation and extraction products or similar carbon containing starting materials such as heavy oils, low temperature oils, extracts of heavy oil sands and the like, which high pressure hot separator is connected in line with the high pressure hydration. The high pressure hot separator is provided with a cyclone separator positioned in the gas/vapour zone in the pressure vessel of the hot separator. The high pressure hot separator has an improved separation efficiency.

HIGH PRESSURE HOT SEPARATOR

The invention relates to a high pressure hot separator for the separation of a head product produced in the high pressure hydration hydrogenation of coals, tars, mineral oils, their distillation and extraction products or other carbon containing starting compositions such as heavy oils, low temperature oils, heavy oil sands or the like. The separator is connected in line after a sump phase reactor for the high pressure hydration. The head product is separated in the hot separator into a gas/vapour phase and a sump product. The high pressure hot separator is constructed from a vertically positioned, cylindrical pressure vessel mantle having a cover and a bottom, heat insulation between the container mantle and an inner cylindrical liner of the vessel, which liner has an integral frusto-conical bottom section, a starting material feeding conduit for the pressure vessel, a discharge conduit for discharge of the gas/vapour phase from the pressure vessel, a sump product drainage conduit and a cooling circuit integrated into the liner for indirect cooling of the hot separator.

Hot separators are known, for example, from installations for liquefying carbon hydration, and are generally constructed of pressure resistant vessels, which have inserts cooled by pipe coils for improved separation and accumulation of a liquid phase in the lower part of the container. The cooling also prevents the separated compounds from carbonizing on the hot walls of the separator, these compounds having a low volatility even at the high temperatures present in the hot separator and including solid or ash forming particles. The lower, cooled section of the separator is conventionally constructed in the form of a funnel through which non-volatile separated components are discharged. However, it has been apparent during operation of such prior art separators, that carbonization problems are frequent in spite of the cooling of the bottom section of the liner by pipe coils. This carbonization may cause an irregular operation of the separator and may even force the operation to be stopped (see "Die katalytische Druckhydrierung von

Kohlen, Teeren und Mineralölen, Springer-Verlag, Berlin/Göttingen/Heidelberg, 1950, page 243 ff.)

Generally, hot separators for the above described use and especially for pressures of up to about 1,000 bar, preferably 150-500 bar, are manufactured in the final geometrical shape and construction of the vessel which is required for high and very high pressure conditions. It is a disadvantage of such vessels of fixed shape that under extreme mass flow variations, which may be the result of, for example, the use of other starting products as those coal varieties or heavy oils suitable for the high pressure hydration, the degree of separation may be substantially lower. This may be the case, for example, in the hydration of heavy oil sand and tar sand extracts, which are characterized, for example, by high aluminum oxide contents from argillaceous earths. These extracts pass as ash promoting components into the head product of the sump phase hydration and, thus, are carried over into the hot separator. Furthermore, the vessels for high and very high pressures are expensive to manufacture and geometrical and constructional changes of such fixed shape vessels for their adaptation to different operating conditions and starting products in order to optimize the degree of separation achieved, would lead to additional cost.

Therefore, it is desirable to construct a hot separator, which is provided with an optimized separating action at relatively low cost, and which has a geometry that is mainly determined by the requirements resulting from its use under high and extremely high pressures. The requirement for improvement of the separating capability of known hot separators is apparent from the fact that in conventional processes at least two hot separators are used, which are connected in series (see German patent DE-PS 933,826), for the production of liquid fuels by catalytic hydration of heavy oils or oil residues in a sump phase hydration under pressure with an immediately following gas phase hydration.

An improved separating action is achieved in a hot separator in accordance with the invention, wherein a cyclone separator is provided in the gas/vapour zone of the hot separator. The cyclone separator has a cylindrical upper section and a conical lower section and an entry conduit for the tangential feeding of the gas/vapour phase into the cylindrical section which gas/vapour phase contains, for example, liquid components carrying solid components. A conical shield is positioned at the axis of the cylindrical section or the conical section and a coaxial central pipe is provided for the removal in upward direction of the gas/vapour phase which has been cleared of liquid components. The central pipe downwardly extends into the cyclone separator beyond the region of the entry conduit and upwardly communicates with the discharge pipe for removal of the gas/vapour phase from the high pressure vessel.

Prior art German patent DE 26 46 605 teaches a system having several reactor stages, wherein an inner cyclone is provided for the retainment of catalyst particles at the head of each reactor stage. The further separation of the catalyst particles is appropriately achieved under process pressure and by way of a cyclone, which is provided in a hot separator in line with the hydration reactors.

Furthermore, German published application 3,405,730 teaches a separator for expansion evaporators of carbon hydration installations as well as a process wherein the suspension produced in the pressure hydration is expanded in one or several steps to a lower pressure before being fed into the separator. The separator is of a cyclone-type construction.

However, it is a disadvantage of such prior art separators that they do not provide a high-grade separating function when they are used in processes and with starting materials in the present invention. However, such a high-grade separator function is absolutely necessary, since a so-called gas phase hydration, which is used for the extraction of products with reform specifications, is

usually carried out immediately after the sump phase hydration and after the removal of the residue phase, which residue phase was separated in the hot, separator. An insufficient separator function would soon lead to a pressure loss in the gas phase hydration, which takes place on a solid bed catalyst. This is due to a deposition on and blocking of the solid bed catalyst by unseparated liquid products which are carried along in the gas/vapour phase and contain solid residues and ash forming components.

The cyclone separator, which is mounted in the gas/vapour zone of a hot separator in accordance with the invention, is only a gas flow controlling apparatus and need not be constructed for high pressures. The optimal construction parameters of the cyclone separator may be calculated according to the process conditions and requirements present.

In a preferred embodiment of the high pressure hot separator in accordance with the invention, the entry conduit of the cyclone separator is provided with a washing arrangement including a wash nozzle and a washing liquid supply conduit. The accumulation of solid material deposits in the entry conduit of the cyclone separator and around the mouth thereof may be substantially prevented with such a washing arrangement.

The starting material feeding conduit for the supply of the head product of a sump phase reactor to the hot separator preferably ends in the gas/vapour zone of the pressure vessel and above the liquid level of the sump product in the hot separator. Furthermore, the conduit is preferably constructed in such a way that the head product discharged therefrom generally downwardly and tangentially impinges on the inner surface of the cylindrical insert of the hot separator.

The sump product drainage conduit of the cyclone separator is preferably submerged with its discharge end below the liquid level of the sump product in the hot separator. It is principally required

that a high negative pressure is maintained in the axial direction of each cyclone. At the high density present in a high pressure hot separator in accordance with the invention caused by the correspondingly high pressure, the negative pressure in the cyclone is much larger than would be expected from conventional applications of cyclone separators. According to calculations made, the cyclone separator would fill from its bottom end. This problem is overcome by a conical shield which is preferably positioned in the cylindrical part and around the axis of the cyclone separator. Appropriate dimensioning of the discharge conduit may substantially prevent a clogging of the conduit by deposited solids.

In another preferred embodiment, the sump product is removed from the conical part of the cyclone separator through a separate conduit, which is connected with an expansion vessel that is connected in line with the hot separator. In this embodiment, the conical part of the cyclone separator may be closed at the bottom. However, the major part of the condensed sump product is still removed from the hot separator through the sump product drainage pipe in the bottom of the hot separator. Only that amount of liquid which is separated in the cyclone separator is removed from the high pressure vessel through the separate conduit, which, for example, extends within the discharge pipe for the gas/vapour phase.

The high pressure hot separator is preferably provided with a liquid level control for either one or both of the vessel and the cyclone. The liquid level may be determined by measuring a pressure difference between a so-called reference conduit extending in the gas/vapour zone of the vessel or the cyclone and a measurement conduit which extends into the bottom section of the conical part of the cyclone separator or the line. Hydrogen is bubbled into the two separate conduits and the pressure difference in these hydrogen supply conduits is recorded. This pressure difference is proportional to the liquid level in the conduits. The hydrogen supply conduits for the liquid level measurement of the cyclone as well as the pipe for the

discharge of the sump product from the conical part of the cyclone separator preferably exit the gas/vapour phase discharge pipe of the pressure vessel through a special lenticular gasket.

Direct introduction of hydrogen containing gases, during measurement of the liquid level, into the liquid sump product accumulated in the lower, conical part of the pressure vessel insert counteracts a hydrogen impoverishment of the sump product, which could otherwise lead to increased coke formation and deposition in the hot separator.

In a preferred embodiment, the vertical, cylindrical section of the insert of the high pressure separator is integral with the conical section of the insert and an adjacent sump product discharge pipe union in the bottom of the pressure vessel.

The cylindrical insert of the pressure vessel is part of a coolant circuit for indirect cooling of the hot separator and is preferably connected to coolant supply and drainage lines in the cover and/or the bottom of the pressure vessel. The insert is preferably constructed of finned pipes as known from conventional boiler constructions. However, the insert may also be composed of regular pipes laterally connected by webs which have been welded therebetween. The tangential impingement of the head product of the sump phase hydration onto the inner surface of the vessel insert provides for a pre-separation of the head product. Thus, an improvement of the separation function of the hot separator is achieved in that an unnecessary whirling up of the liquid accumulated in the bottom of the hot separator by condensed liquid components falling into the liquid from a certain height is substantially prevented.

The high pressure hot separator of the invention may be provided with a wear resistant coating made, for example, of tungsten carbide or wear resistant ceramic, especially when the head product of the

sump phase hydration contains highly wear intensive mineral components, for example, aluminum oxide of argillaceous earths. This may be the case when oils from tar sands are used as the starting products of the high pressure hydration. The wear protection coating may be provided only in especially wear intensive zones or over the whole interior surface of the hot separator.

The invention will now be further described by way of example only and with reference to the following drawings, wherein

Figure 1 is a vertical cross-section through a high pressure hot separator in accordance with the invention;

Figure 2 is a horizontal cross-section along line A-A through the embodiment shown in Figure 1;

Figure 3 is an enlarged partial horizontal cross-section through the cyclone separator of the embodiment shown in Figure 1; and

Figure 4 is an enlarged longitudinal cross-section through the gas/vapour phase discharge pipe of the embodiment shown in Fig. 1.

In the preferred embodiment of a high pressure hot separator in accordance with the invention, the hot separator includes a cylindrical, vertically positioned vessel mantle 11 having a flanged end of increased wall thickness. A cover 12 and a bottom 13 are respectively securely bolted to the mantle 11. A heat insulation 14 is provided along the inner surface of the pressure vessel mantle 11, cover 12 and bottom 13. A non-supporting insert 18, which is conically tapered at its lower end, is located inwardly and against the heat insulation of the pressure vessel mantle 11. The conical bottom 18a of insert 18 is at its lower end integral with a sump product drainage pipe 5. Head product of a sump phase hydration carried out in a sump phase reactor is supplied into the high pressure vessel through a starting material feeding conduit 1, which extends

through cover 12. The head product is separated in the high pressure hot separator into a sump product, which is removed from the separator through sump product drainage pipe 5, and a gas/vapour phase, which is removed from the hot separator through a gas/vapour discharge pipe 3 that also extends through cover 12. Under the pressure and temperature conditions present in the high pressure hot separator, the gas/vapour phase is cleared of liquid components, which may also contain residue or ash producing components, and of condensed liquid components from the hot separator. The starting material feeding conduit 1 is bent at its discharge end in such a way that the head product of the sump phase reactor, which contains liquid and residue components, tangentially downwardly impinges on the inner surface of the insert 18 of the pressure vessel mantle 11 just above the liquid level in the hot separator. This liquid level is maintained constant through appropriate measurement and control apparatus. The measurement and control apparatus are supplied with the required data by a temperature sensor 16 and level sensors 9.

A cyclone separator 4 is mounted to cover 12 co-axial with discharge pipe 3 of the gas/vapour phase and within the gas/vapour zone of the high pressure hot separator. The cyclone separator 4 includes conventional components, especially an intake conduit 2, an upper, cylindrical section 4a, a lower conical section 4b and a central pipe 4c. The central pipe 4c is mounted to the upper end of cylindrical section 4a and is connected to discharge pipe 3. The central pipe 4c downwardly extends in the cylindrical section of the cyclone separator and ends below the area of entry of the intake conduit 2. This construction substantially prevents a forced mixture or a short circuit mixing of a first process stream, which enters the cyclone separator through intake conduit 2 and contains liquid components, with a second process stream of the "dried" gas/vapour phase entering the lower end of central pipe 4c for discharge through discharge pipe 3. A washing liquid supply line 7, which supplies an appropriate washing liquid to a washing liquid nozzle 6 for the cleaning of the entry conduit 2, extends within discharge pipe 3. The

drainage pipe 10 of the lower, conical section 4b of the cyclone separator 4 is submerged with its lower end in the liquid accumulated at the bottom of the high pressure vessel.

The discharge pipe 3 and the measurement connections and product pipes extending therethrough are illustrated in greater detail in Figure 4. The reference numerals in Figure 4 define the same parts as the corresponding numerals in Figures 1 to 3. Figure 4 shows a special lenticular gasket 17 through which extend the washing liquid supply line 7 and the cables 15 for the liquid level measurement. A suction pipe 20 may also extend through the discharge pipe 3 for removal of sump product from the cyclone separator, when the cyclone separator is equipped with a closed conical bottom section.

Conical shield 19 which is co-axial with the conical section 46 of the cyclone separator 4 shields the submerged drainage pipe 10 of the cyclone separator from the vacuum present along the axis of the cyclone separator.

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. A high pressure hot separator for the separation of a head product of a high pressure hydration into a gas/vapour phase and a sump product, which head product is produced in a process for the high pressure hydration of coal, tar, mineral oil, their distillation and extraction products or similar carbon containing starting materials such as heavy oils, low temperature oils, extracts of heavy oil sands and the like, which high pressure hot separator is connected in line with the high pressure hydration, comprising

a vertically positioned cylindrical pressure vessel mantle having a cover and a bottom, a heat insulation on the inner surface of the cylindrical pressure vessel, a cylindrical insert having a lower conical section, a starting material feeding conduit for supplying the head product into the pressure vessel, a discharge pipe for removing the gas/vapour phase from the pressure vessel, a sump product drainage pipe and a cooling circuit incorporated into the insert for indirectly cooling the pressure vessel, and a cyclone separator provided in the gas/vapour zone of the high pressure hot separator, the cyclone separator having a cylindrical upper section, an intake conduit for permitting tangential entry of the gas/vapour phase into the cylindrical section, which gas/vapour phase includes liquid components containing solids, a conical, lower section, a conical shield positioned around the axis of one of the cylindrical and conical sections, a central pipe co-axial with the cylindrical section for upwardly discharging the gas/vapour phase cleared from liquid components, the central pipe extending downwardly beyond a region of entry of the intake conduit into the cylindrical section and being upwardly connected with the discharge pipe of the gas/vapour phase.

2. A high pressure hot separator as defined in claim 1, wherein the intake conduit of the cyclone separator is provided with a washing arrangement including a washing liquid supply pipe and a washing

liquid nozzle for substantially preventing a deposition of solid materials in and around the intake conduit.

3. A high pressure hot separator as defined in claim 1, wherein the starting material feeding conduit extends in the gas/vapour zone of the pressure vessel and ends above the liquid level of sump product accumulated in the hot separator, the starting material supply pipe being further constructed in such a way that the starting material substantially tangentially and angularly downwardly impinges on an inner surface of the cylindrical insert.

4. A high pressure hot separator as defined in claim 1, wherein a lower end of the drainage pipe for removing the sump product from the conical section of the cyclone separator is submerged in the liquid sump product accumulated in the hot separator.

5. A high pressure hot separator as defined in claim 1, wherein the sump product is removed from the conical section of the cyclone separator by a suction pipe connected with an expansion vessel, which expansion vessel is connected in line with the hot separator.

6. A high pressure hot separator as defined in claim 1, wherein the conical section of the cyclone separator is downwardly closed.

7. A high pressure hot separator as defined in claim 1, wherein the cyclone separator is provided with a liquid level measurement.

8. A high pressure hot separator as defined in claim 1, wherein the conical bottom of the insert of the hot separator is provided with a gas supply pipe for supplying hydrogen containing gases into the liquid sump product.

9. A high pressure hot separator as defined in claim 1, wherein the pressure vessel mantle is reinforced at an upper and lower flange zone respectively.

10. A high pressure hot separator as defined in claim 1, wherein the conical bottom of the insert ends in the sump product discharge pipe of the hot separator.

11. A high pressure hot separator as defined in claim 1, wherein the insert of the pressure vessel includes at least one of finned pipes and regular pipes which regular pipes are welded together by means of intermediate webs, for indirect cooling of the pressure vessel.

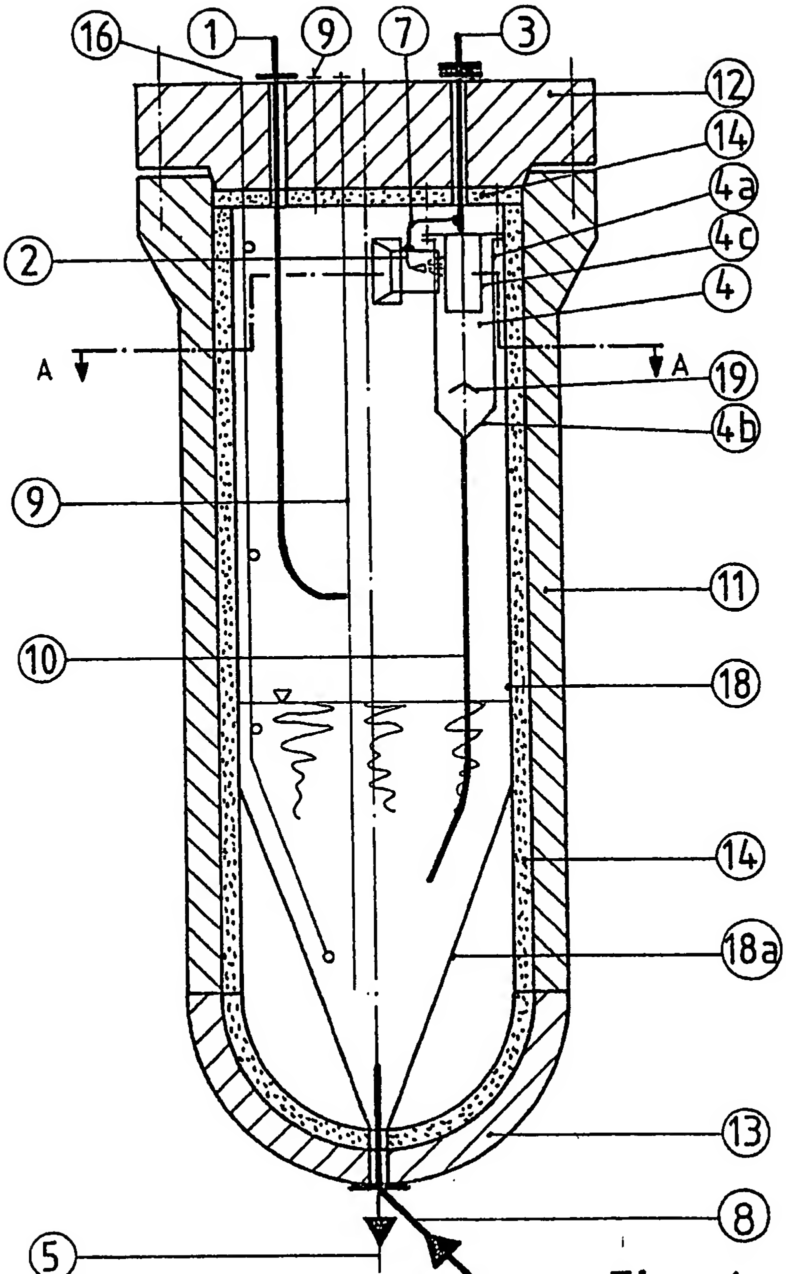

Fig. 1

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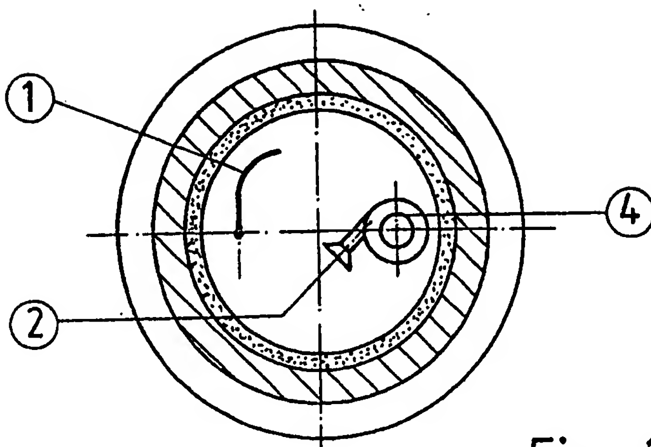

Fig. 2

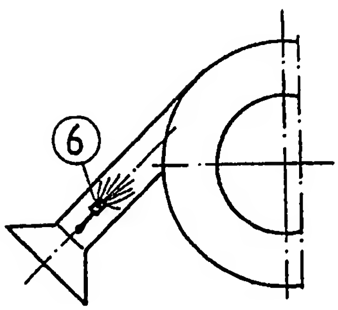

Fig. 3

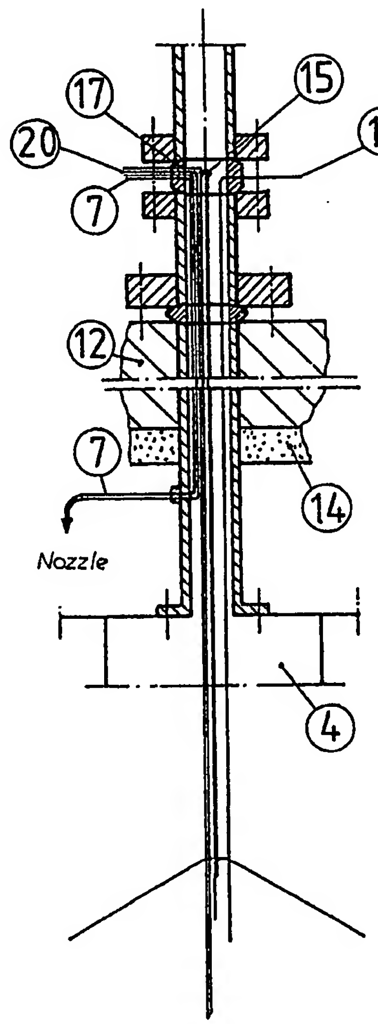

Fig. 4

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